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Second Topical Meeting on Crystal Growth Mechanism

Crystallization of Amorphous Silicon Irradiated With Synchrotron Radiation

43070731A Tokyo MONBUSHO KAGAKU
KENKYUHI HOJOKIN KENKYUKAI HOKOKU
RONBUSHU in English 17, 18 Jan 90 pp 41-44

[Article by Fumio Sato and Jun-ichi Chikawa: "NKH Science Research Laboratories, 1-10-11 Kinuta, Setagaya-ku, Tokyo 157; National Laboratory for High Energy Physics, Tsukuba, 305, Japan"]

[Text] Selected papers from Proceedings of the Second Topical Meeting on Crystal Growth Mechanism held 17, 18 Jan 90 in Izu Nagaoka, supported by the Grant-in-Aid of Science Research on Priority Areas From the Ministry of Education, Science and Culture]

Abstract

Amorphous silicon deposited on a single crystal wafer was irradiated with synchrotron radiation at room temperature for 72 hours, and its effect on crystallization was investigated by Raman spectroscopy. After the irradiation, no difference between irradiated and non-irradiated portions was observed. After annealing at 600°C for 1 hour, a large difference between both the portions was recognized by Raman spectra due to polycrystalline silicon. The irradiation-enhanced crystallization may be attributed to Si-Si bond-breaking by the irradiation.

Introduction

Photons provide a high energy to assist crystal growth and etching, e.g., 1 eV, photon energy in the infrared region corresponds to thermal energy per atom at 10⁴ K. Whereas, the latent heat per atom corresponding to bonding energy at the melting point is generally about 0.5 eV or less. To break a bonding or to incorporate an atom into the crystal lattice, higher activation energy must be overcome, and energy larger than several eV is required. Recently, synchrotron radiation has been employed for such purposes. In general, photon beams are used during crystal growth or etching. In the present work, to investigate radiation effect irradiation and growth were made separately; irradiation was made at a temperature far lower than the growth temperature, and then growth at an enough high temperature.

Experimental

Specimens were 3000-A-thick amorphous silicon (a-Si) deposited on silicon single crystal wafer by the plasma CVD method. The a-Si films contain hydrogen at about 10 atomic percent. An a-Si film was irradiated for 72 hours in high-vacuum with synchrotron radiation (SR) from the storage ring operated at an electron energy of 2.5 GeV and average current of 150 mA in the SR facility "Photon Factory". The total irradiation power was

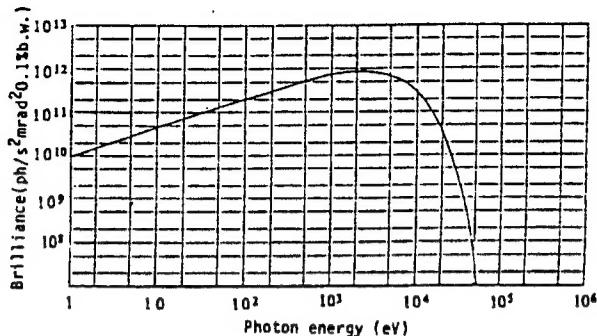


Figure 1. Spectrum of synchrotron radiation from the electron storage ring at the Photon Factory (Electron energy 2.5GeV, average beam current 150mA)

about 24 W/cm², and the a-Si film was kept at 20°C during the irradiation by using a water-cooled specimen holder. The spectrum of the photon beam used is shown in Figure 1. The irradiated area is 5×5 mm of the specimen (15×20 mm). The total number of photons was estimated to be 8×10²¹ photons/cm². Assuming that the average absorption rate per monatomic layer is 3×10⁻⁶, the number of photons absorbed by the amorphous specimen film was estimated to be 5×10¹⁹ photons/cm², and it turns out to be 10 photons absorbed per Si atom.

Crystallization of the a-Si film was investigated by the laser-Raman Spectroscopy, and hydrogen contents in the films was measured by SIMS.

Results and discussion

Raman scattering intensities were measured for the irradiated and non-irradiated parts of the specimen as shown in Figure 2(a) and (b), respectively. The spectra give no indication of crystallization, and no difference between both the parts is recognized. Even if crystallization occurs, crystallites far smaller than the wavelength of the primary laser beam (=5145 Å) cannot be detected. To grow them, therefore, this specimen was annealed at 600°C for 1 hour, and then the same measurement was carried out. The results are shown in Figure 3. The sharp peak at a wave-number shift of 522 cm⁻¹ is due to polycrystalline silicon. The twice higher peak for the irradiated part in Figure 3(a) indicates that irradiation causes much faster crystallization than the non-irradiated part. After the annealing, the irradiated part can be recognized clearly with the naked eye as seen in Figure 4 [photograph not reproduced]. This fact shows that SR irradiation enhances crystallization in the following anneal.

The latent effect of irradiation was kept stably to be revealed even by annealing 6-months after the irradiation. By comparing the observed spatial variations of the Raman intensity for the irradiated part and the spectral distribution of the SR in the vertical direction, it was

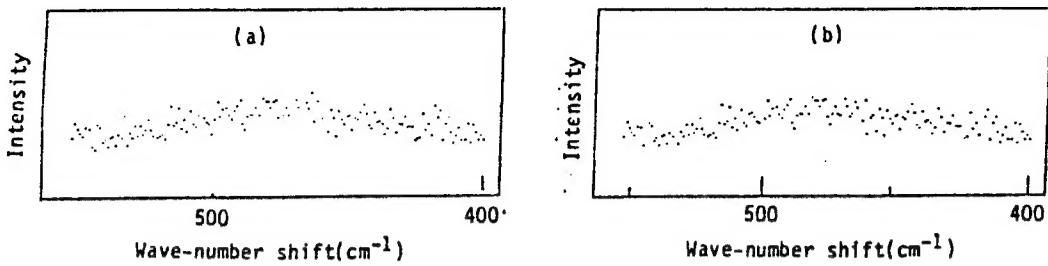


Fig. 2 Raman spectra measured for the amorphous silicon after irradiation with the synchrotron radiation
(a) irradiated part (b) non-irradiated part

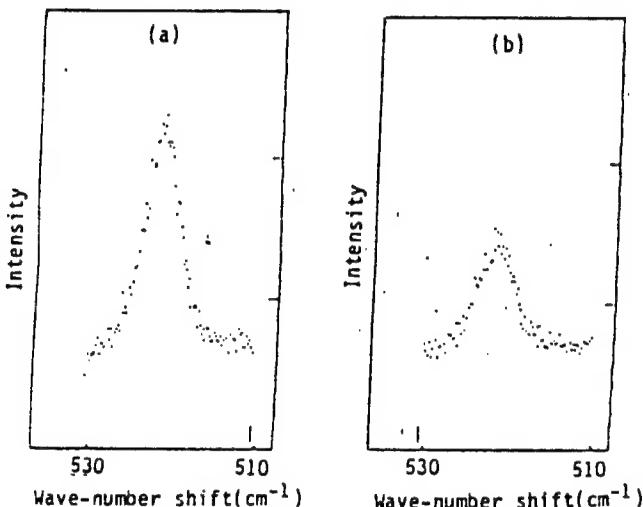


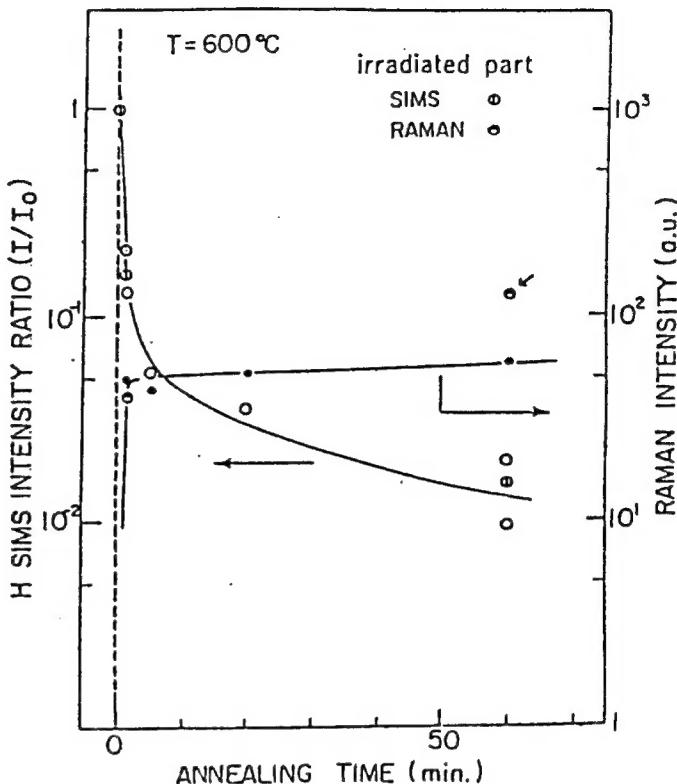
Figure 3. Raman spectra measured after annealing the irradiated amorphous silicon at 600°C for 1 hour. (a) irradiated part. (b) non-irradiated part. The peak at a wave-number shift of 525cm⁻¹ is due to crystallization.

found that photons in both the soft x-ray and VUV regions are responsible for the enhanced crystallization.

Figure 5 [photograph not reproduced] shows optical micrographs of the annealed specimen. In both the irradiated and non-irradiated parts, grains with sizes of 3 to 4 μm are seen dispersedly. It was found by Raman microspectroscopy that the Raman peaks in Figure 4 are due to such grains, and the matrix between grains are still amorphous. In comparison of both the parts, it should be noted that grains are seen black in the irradiated part and white in the non-irradiated part, although densities and sizes of grains are similar between both the parts. White and black grains seem to be polycrystal- and single-crystal-like, respectively.

Variations of hydrogen contents in the specimens were measured by SIMS, as seen in Figure 6. The hydrogen contents in both the irradiated and non-irradiated parts were the same before annealing and decrease rapidly owing to out-diffusion to 1/20 of the initial value within a few minutes. Since the content variations in both the parts were similar, the observed difference in the Raman peak intensity between the irradiated and non-irradiated parts cannot be attributed to hydrogen. It was observed that the Raman peak at 522 cm⁻¹ increases within a few minutes from the initiation of annealing as seen in Figure 6, and the difference in the Raman peak between the irradiated and non-irradiated parts was found to be produced after the hydrogen diffuses out. Therefore, the mechanism for the irradiation-enhanced crystallization may be explained by assuming that Si-Si bond-breaking is caused by the inner shell excitation.

Fig. 6. Variations of hydrogen contents and Raman peak intensity at 522 cm^{-1} with annealing time at 600 °C. I_0 is the initial SIMS intensity
 ○ for 20-hr irradiation
 ● for 72-hr irradiation



Preparation of High T_c Bi-Sr-Ca-Cu-O Films on MgO Substrates by Liquid Phase Epitaxial Method

43070731B Tokyo MONBUSHO KAGAKU
KENKYUHI HOJOKIN KENKYUKAI HOKOKU
RONBUSHU in English 17, 18 Jan 90 pp 103-106

[Article by Hiroyuki Takeya and Humihiko Takei:
"Institute for Solid State Physics, University of Tokyo,
7-22-1 Roppongi, Minato-ku, Tokyo 106"]

[Text] Abstract

Thin Films of high T_c superconductor Bi₂Sr₂CaCu₂O_x were prepared on MgO (100) substrate by a liquid phase epitaxial method. Several kinds of fluxes were applied for preparation of Bi₂Sr₂CaCu₂O_x thin films, and KCl was found to be the best among them. The film was made of two phases, Bi₂Sr₂CaCu₂O_x and Bi₂Sr₂CuO_x. The on-set T_c in the superconducting transition was at 95 K in an as-grown state, but zero resistivity was not observed over 50 K.

The new superconductor on Bi-Sr-Ca-Cu-O (BSCCO) system found by Maeda et al.¹⁾ has notable advantages in comparison with YBa₂Cu₃O_x (YBCO) as follows. (1) BSCCO includes higher T_c phase than YBCO. (2) BSCCO does not easily make oxygen vacancy around the Cu-site which directly influences superconductivity in YBCO case. (3) BSCCO is chemically stable against water. They are very important from the standpoint of practical use. Many studies on wire-drawing and film-making of BSCCO has

been carried out with an intention of applying superconductor. Especially thin films have a high potential application for electronic devices.

Although there have been reported several successes in producing BSCCO thin films using rf-magnetron sputtering^{2,3)}, electron beam evaporation⁴⁾ or chemical vapor deposition⁵⁾, no discussion was included about the epitaxial direction to a substrate or a problem of a mismatching between films and substrates. The present study is a trial to make thin films by a liquid phase epitaxial (LPE) method using flux. The LPE method has several advantageous points: (1) The LPE method, different from sputtering or electron beam evaporation, can be performed in an atmospheric pressure. This makes a reduction of oxygen defects in the film. (2) It is easier to obtain information of film formation process. The LPE method is carried out in a condition near an equilibrium state, and the lattice-mismatch of substrate with a film becomes clear. (3) There exists much hope of practical use in the LPE method because of its high growth rate.

In this study, various kinds of fluxes are used for preparing films on a MgO(100) substrate. It was found that a Bi₂Sr₂CaCu₂O_x film was grown only from a KCl flux. The as-grown film showed superconducting transition at 95 K, on-set T_c, but had residual resistivity at 50 K by 1/10 to 95 K. The film oriented along the c-axis of MgO with the same orientation, whereas no orientation was found in the a-b plane. It might be caused by the mismatching of the lattice parameter about 10 percent between MgO and Bi₂Sr₂CaCu₂O_x.

A sintered $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ powder was prepared as follows. Bi_2O_3 , SrCO_3 , CaCO_3 , CuO which were reagent grade were mixed at the atomic ratio $\text{Bi:Sr:Ca:Cu}=2.2:1.2$ and were heated two times at 780°C and 830°C for 12 h. The sintered material mixed with flux were melted in a Pt crucible using a rf furnace. Bi_2O_3 , CuO , V_2O_5 , PbO , KCl , KF and NaCl were used as a flux. A substrate crystal ($5 \times 15 \times 1 \text{ mm}^3$) hung by Pt wire was put into a solution at 1200°C for 1 min. The film was observed by a scanning electron microscope (SEM) and analyzed by an electron probe microanalyzer (EPMA). X-ray probe analyses were also carried out by a diffractometer, and Laue and precession cameras. The resistivity were measured by a conventional dc-four probe method.

At first, we tried no flux condition: $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ without any flux was melted at 1200°C , a MgO substrate was dipped into the melt and then pulled out after 1 minute. The film formation was not observed between 1200°C and the melting point of this system. Next, we tried a self flux condition: $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ mixed with a self flux, Bi_2O_3 or CuO at a mole ratio 1:1 respectively, was in the experiment. In the case of using Bi_2O_3 as a flux, a film was formed on the MgO (100) substrate. X-ray diffractometric analyses showed that only the $001(1=2n)$ diffraction lines of $\text{Bi}_2\text{Sr}_2\text{CuO}_x$ ($c=24$ angstrom) appeared. This means that CuO is not effective in making $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ films. Thirdly, we used V_2O_5 or PbO as a flux, which was added 7 times (mole ratio) of sintered $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$. The film of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ composition was not observed under any temperature conditions.

Finally, we used alkali halides such as KF , NaCl and KCl ⁶ as a flux. Among them, KCl was reported to be a good solvent material for growing a large single crystal of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ ⁷. The film formation was not observed on the cases of KF and NaCl fluxes where the ratio of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ and KF/NaCl was 1:1.

The black, conductive films were formed when KCl flux was used at a mole ratio of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x:\text{KCl}=1:200$ or 1:7. The film produced from the first ratio at 1200°C was judged to be $\text{Bi}_2\text{Sr}_2\text{CuO}_x$ by its diffraction pattern, as shown in Figure 1, because only the $001(1=2n)$ lines of $\text{Bi}_2\text{Sr}_2\text{CuO}_x$ were observed in it. In the case of 1:7, the film of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ was successfully grown at 1200°C . The diffraction pattern of the film is shown in Figure 2. There are recognized the major phase as $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ and the minor phase as $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ in the figure. Although the $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ film was prepared only using KCl flux, there might remain some good conditions for the other fluxes that have not been searched yet. Figure 3 [photograph not reproduced] shows a typical SEM photograph of the film prepared from the solution using KCl flux. The film was made of two parts: the bright area (A) and the dark area (B). EPMA determined that the part A was Bi-Sr-Ca-Cu oxide with the composition of $\text{Bi:Sr:Ca:Cu}=2.4:2.3:0.7:2.0$, and the part B MgO , respectively. The area A is composed of platy grains, where the grain size is $10-50 \mu\text{m}$ in diameter. The film thickness was determined to be about $1 \mu\text{m}$ from the cross-section in SEM photographs.

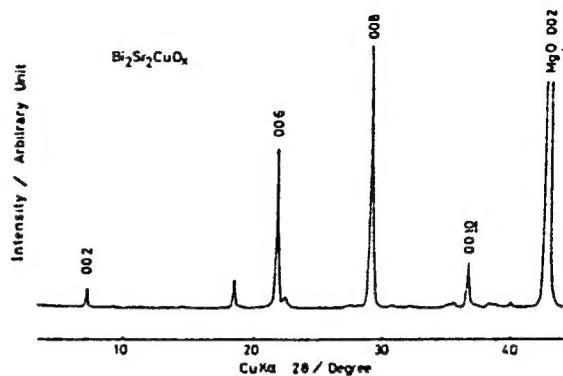


Figure 1. The ray diffraction pattern of the film grown from the KCl flux at the ratio of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x:\text{KCl}=1:200$.

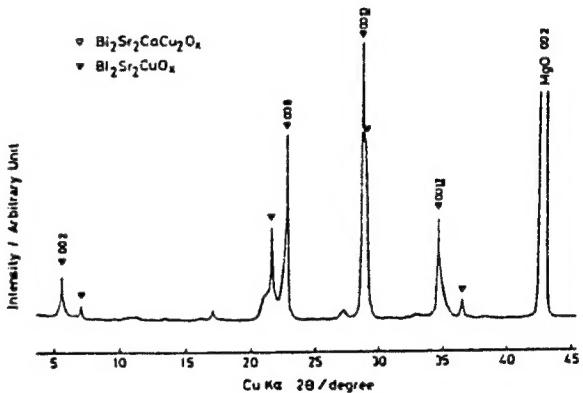


Figure 2. The X-ray diffraction pattern of the film grown from the KCl flux at the ratio of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x:\text{KCl}=1:7$.

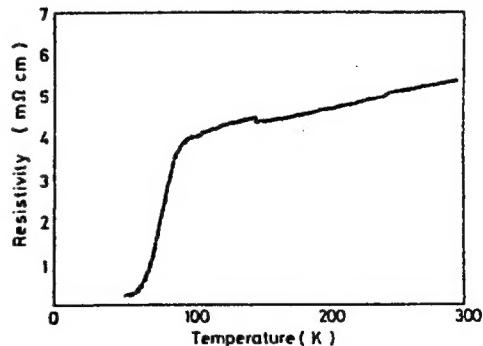


Figure 4. The temperature dependence of the resistivity for the film grown from the KCl flux at the ratio of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x:\text{KCl}=1:7$.

The electric resistivity in the as-grown state measured by a conventional dc-four probe method is shown in Figure 4. Though the on-set T_c was observed at 95 K, zero resistivity was not above 50 K. It might be caused by the incomplete dense film or the existence of the sub-phase, $\text{Bi}_2\text{Sr}_2\text{CuO}_x$.

The crystallographic direction between the film and the substrate was determined. Precession photographs of films were taken together with the substrate MgO which was shaved off to about 100 μm thick. A typical photograph was shown in Figure 5a [photograph not reproduced] where the x-ray beam passed perpendicularly through the film. Figure 5b [photograph not reproduced] is an expanded photograph of Figure 5a, in which we can see the ring pattern which corresponds to the 200 diffraction of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$. Figure 6a [photograph not reproduced] is a photograph which was taken after rotating the substrate to the horizontal position to the x-ray beam.

Figure 6b [photograph not reproduced] is an enlargement of Figure 6a. The spots correspond to 008, 0010, 0012 of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ were observed in it. From the figures 5b and 6b, the film was formed as the c-plane-oriented polycrystal along the (100) plane of the MgO substrate. This may be caused by the large mismatching of the lattice parameters between the film and the substrate. The lattice parameter a of MgO is 4.213 angstrom⁸⁾ and that of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ is 5.400 angstrom⁹⁾. The packing arrangement of anions is important in the discussion about mismatching. Based on the lattice parameters and the structure of Tarascon et al.¹⁰⁾, the distance between the first nearest oxygens in each layer and the square root of 2 times the distance between them are calculated and shown in Table I. The misfit parameter f is calculated by the equation:

$$f = d_s - d_e / d_c$$

where d_s and d_e mean the 0-0 distance of the substrate and the film, respectively, at the contact plane. It is most likely that the contact plane of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ to MgO (100) is the (001) plane of the (Ca,Sr)O layer, the structure of which is the same NaCl-type as that of the substrate MgO . The least value of f is 10.3 percent from the combination of MgO :4.213 angstrom and SrO :3.818 angstrom. It is suggested from this study that 10.3 percent is too large to grow a single crystal film of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ on MgO (100) near the equilibrium condition.

Table 1. The distance between the oxygens of each layer in the MgO (100) and $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (100) planes.

Layer	Distance between the first nearest oxygens (angstrom)	\times the square root of 2 (angstrom)
Mg-O	2.979	4.213
(Ca,Sr)-O	3.818	5.398
Bi-O	3.818	5.399
Cu-O	2.700	3.818

Acknowledgments

The authors wish to thank Dr. Tsuyoshi Tamegai for the measurement of low-temperature resistivities. Thanks are also due to Mr. Tsuneo Kitazawa for his technical assistance.

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New Plastic Converts Light Directly to Heat

90P60003A East Berlin RADIO FERNSEHEN ELEKTRONIK in German No 4, Apr 90 p 205

[Text] Japanese researchers have developed a new plastic industrial polymer which can convert light directly into heat energy. When the material is irradiated with visible or ultraviolet light for an interval of at least 1.5 minutes, it generates 420 joules/gram of heat energy, sufficient to boil water. The functional dependence of the polymer's design coefficient upon the irradiation is a material property which has potential for application in the manufacture of optical memories for reading, writing and erasing data.

New Developments in Biotechnology**Marine Microorganism-Based Production of Active Substances**

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 17

[Report by Yoshiro Okami, Institute of Microbial Chemistry]

[Text] 1. Purpose

The marine environment differs from the land environment. For instance, the state of seawater, current, and the sea bottom differ from the inshore shallow sea to the steep, deep sea over a continental shelf. In the deep sea the environment is dark and dystrophic, the temperature is low, and pressure is high. The seaweed, fish, and shellfish that survive in such an environment are unique. Body components and metabolic secretions that are not found in land organisms have been obtained from various seaweed, fish, and shellfish. It is possible that previously unknown substances may be found in marine microorganisms. New and useful bioactive substances may be obtained by taking microorganisms specific to the ocean and testing them for antibacterial, antitumor, or physiologically active substances. Compared with seaweed, fish, and shellfish, it is easy to artificially culture and separate microorganisms and reproduce useful substances from them. This characteristic is advantageous in mass production of medical substances. Such research is useful in developing knowledge of marine resources and the ocean and in finding uses for microorganisms. This report outlines this research.

2. Method

Samples of seawater, sea sludge, and marine organisms are taken from the sea. Microorganisms are then separated and cultured in various artificial environments (for instance, medium temperature) to detect biological activities (such as the activities of enzymes and pathogenic organisms). Active substances are extracted and purified from the culture by various physical and chemical methods to determine their chemical structures and to measure their physical, chemical, and biological qualities. On the basis of this information, specific properties of marine microorganisms, such as pressure resistance, salinity resistance, and ecological relationships, are studied.

3. Results

A method for separating and culturing various microorganisms from samples taken from the shallow sea and the deep sea (to a depth of 2,000 meters) was established. It was determined that some marine microorganisms that require seawater for growth and production of active substances produce bioactive substances with specific chemical structures. For instance, some enzymes, such as Aplasmomycin which prevents the reproduction of *Plasmodium malariae* and Bisucaberin

which changes cancer cells into cells that are readily dissolved in phagocytes, have been obtained.

Development of Novel Sensors Using Marine Microorganisms, Luciferase

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 19

[Report by Eiichi Tamiya and Ikuo Karube, Research Center for Advanced Science and Technology, University of Tokyo]

[Text] 1. Development of Sensors Using Marine Micro-organisms

The authors and their coworkers have developed many sensors using microorganisms. With these sensors, they have measured target substrates by using respiratory activities and metabolic products of microorganisms as indexes. They have developed a sensor using microorganisms based on the luminescence of marine photobacteria. Photobacteria respond to various substances and change their luminous strength. In this experiment, luminous strength was detected by photomol [phonetic], and the amount of particular substances was measured. First, they surveyed the response of *Photobacterium phosphoreum* MT10204 to glucose and found that its luminous strength increases with glucose. The photobacterium was then absorbed and immobilized on a nitrocellulose membrane filter and put in a flow cell, which was installed at the detection part of the photomol. With this sensor, they measured the amount of glucose and found that the time of response was 2 minutes and that glucose in concentrations from 0.05-0.55 mm could be measured.

Then, they measured the amount of harmful substances with this sensor. First, they surveyed the response to benzalconium chloride and SDD and found that the addition of harmful substances decreases its luminous strength. The reaction inhibiting luminescence by these substances were irreversible. In addition, they found that each substance can be quantitatively measured by comparing the strength of response to glucose after the addition of a substrate or the decreased values of baselines. With this system, they tried detecting agricultural chemicals. They found that five kinds of agricultural chemicals, including herbicides that inhibit plant photosynthesis, were detectable with this system and that agricultural chemicals were irreversible inhibitors of luminescence. Next, they applied this system to the detection of heavy metal ions. They studied arsenic, lead, copper, and chromium. A reversible decrease in luminous strength with six-valency chromium was observed. A very stable corelationship was obtained between the decrease in luminous strength and the addition of potassium dichromate, which makes it possible to quantitatively measure chromic acid.

2. Development of a Gene Detection System With Luciferase Used as an Index

With the progress in recombinant DNA technology, genetic studies have been increasing. In these studies, foreign genes are inserted into various cells, from lower to higher organisms, to survey expression and behavior of the genes. Effective expression of foreign genes is very important in these cases. Because of its high quantum yield, the luminescent reaction of luciferase has drawn attention. The authors considered using this reaction as a marker for gene expression. Using cells with luciferase genes inserted, they measured luminous activity with a highly sensitive photo-detection system and evaluated gene expression. First, a plasmid vector pRSV containing the luciferase gene was inserted into *E. coli* strain HB 101. These plasmid-inserted bacteria were liquid-cultured, and *E. coli* cells were collected on a nitrocellulose filter without breaking cells. After they were sprayed with a specified amount of substrate luciferin, luminescence was detected. The luminous reaction was very fast: the peak was obtained by 0.3 second, and the response then decreased. This luminescent pattern was the same as that formed when substrate luciferin was injected after sufficient ATP was added to purified luciferase. The luminous reaction of *E. coli* at each bacterial age was measured next. Bacteria in their logarithmic later phase showed the highest luminous activity. With bacteria in logarithmic later phase, the relationship between the number of bacteria and the unit luminous amount was studied. It was determined that as many as 10^3 bacteria were measured by this method. Considering the rate of luminescent light entering the photo-accepting part of a photomultiplier tube, it was estimated that at least 10^2 - 10 bacteria were measured correctly.

The rate of luminous reaction was then measured with broken bacteria. The amount of luminescence was less than for unbroken bacteria for both peak value and total luminescence. This suggests that the condition of luciferase present in a cell is very important to the gene's expression of activity. Luciferase genes were inserted into cyprinodonts eggs, and the eggs were fertilized and hatched. Five out of 17 eggs inserted with DNA showed a significant amount of luminescence. Their expression activities varied, suggesting that the luciferase gene is present in the cell nucleus of cyprinodont in various forms.

Biodegradable Plastics (I)—Preparation of Degradable Polyethylene, Its Degradability

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 122

[Report by Akira Iwamoto, Yutaka Tokiwa, and Kiyoshi Takeda, Fermentation Research Institute, the Agency of Industrial Science and Technology JSP Co.]

[Text] 1. Purpose

Recently, some Western countries have begun to prohibit or restrict the use of plastics. In the United States,

degradable plastics produced by mixing polyethylene and starch have been put into practical use. In evaluating degradability, however, there are problems with reproducibility, quantitative measurement, and simplicity. Therefore, the authors prepared a degradable plastic consisting of an aliphatic polyester and a polyethylene that is thermoplastic and biodegradable. They then studied an evaluation method for its degradability.

2. Method

Polycaprolactone (PCL), which has a high molecular weight, was used as an aliphatic polyester. A low-density polyethylene (LDPE) was used as a polyethylene. For mixing PCL and LDPE, a mulling and extrusion machine (manufactured by CSI Inc., USA, Type CS-194A) and an inflation film production facility (produced by Modern Machinery) were used. Degradability was evaluated by reacting purified lipase of *Rhizopus arrhizus* (produced by Sigma, Inc.) with the mixture and by measuring water soluble organic carbon (TOC) produced in the reaction solution, the turbidity of the solution, and the reduction in the weight of the samples.

3. Results

The mixing ratio PCL/LDPE was 90/10-10/90 (wt %) by the mulling and extrusion machine and 30/70-10/90 by the film-producing facility. The transparency of the samples decreased as PCL content was increased. The amount of produced TOC after the reaction with lipase changed in proportion to the time for reaction, the enzyme concentration, and the weight of the sample. The effect of the mixing ratio was high degradability up to 60 (wt %) PCL. TOC production based on PCL degradation was drastically reduced with 30 (wt %) PCL or less. It was found that degradation was inhibited by LDPE. From this study, it was determined that this enzymic method was advantageous for developing and evaluating degradable plastics because this procedure was performed quickly and quantitative measurement was easily carried out.

Biodeodorant: Gaseous Substrate Consumption by Immobilized Microorganisms

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 124

[Report by Junichi Koizumi and Tadahiro Mori, Agricultural Department Shimane University]

[Text] 1. Introduction

Smells, particularly offensive smells, have physical and emotional effects in our daily life and working environments. In some industrial areas, physical and chemical equipment to treat smells have been adopted and operated. In addition, there is a demand for the development of a biological deodorant system. This report presents the studies on a system for removing offensive smells caused by sulfur compounds, including the screening of microorganisms functioning mainly in this system, the

development of a carrier that immobilizes and retains microorganisms in a gas distribution system, and the efficiency of a biodeodorant consisting of a dry deodorant column produced by combining them.

2. Microorganisms and Materials for the Carrier

Microorganisms were collected from sludge in under-water tubes and air-seasoned tanks. From these microorganisms, those that metabolize hydrogen sulfate were screened by batch test with air containing 10 ppm (v/v) of hydrogen sulfate used as its substrate. Most of the isolated microorganisms were bacteria, but a kind of Ascomycetes (OMSOfl: genus was not identified) having metabolic activity for hydrogen sulfate was found. The rate of hydrogen sulfate consumption by OMSOfl was 17.7 mg H₂S/g dry microbe x h⁻¹ (H₂S concentration in the gaseous phase was 10 ppm, and the total hydrogen sulfate transfer capacity coefficient was approximately 0.5 s⁻¹). Because fungi can survive under lower humidity than bacteria, an immobilizing carrier of the structure stated below was produced, and an apparently dry deodorant column was established. A piece of poly-acrylic resin, a water absorbant, was covered with foamed urethane, an ester, which was swelled by AT medium. Then OMSOfl was immobilized on the urethane.

3. Biodeodorant

The immobilized OMSOfl was filled in a glass column, to which air containing 28 ppm H₂S with its holding time of 2 minutes was distributed. During a 20-day period, the biodeodorant showed nearly 100 percent removing efficiency.

Antibiotics Production by Deodorant Microorganisms To Inhibit Plant Disease

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 125

[Report by Yasuaki Hashimoto and Yoshiyuki Ohta, Faculty of Applied Biological Science, Hiroshima University]

[Text] 1. Purpose

The authors have found deodorant microorganisms that deodorate livestock waste and excessive sludge in a short period. This time, they have separated deodorant microorganisms having antibiotic activities to shirobakterebyo [phonetic] and studied their properties and their in vivo antibiotic activities.

2. Method

Strains possessing antibiotic activities to *Xanthomonas oryzae* from 400 strains of deodorant microorganisms by Cross-streak method. Screened strains were cultured while stirring in a liquid medium consisting mainly of 10 percent water extracted from pigs' droppings. The

potency of supernatant of the culture medium was examined by the Paper-Disk method. In addition, the constituents of medium culture conditions were studied to raise antibiotic activities. The temperature, time, pH changes during culture, carbon source, nitrogen source, and addition of metal salts were also studied. Then, they experimented inhibiting effects on the rice plant shirobakterebyo. In this experiment, bacterial suspension of *Xanthomonas oryzae* was inoculated by injection, and the supernatant of the culture solution was sprayed as a drug.

3. Results

Seven strains of deodorant microorganisms having antibiotic activity have been screened from about 4,000 strains. *Streptomyces* sp. N112 showing the highest activity were selected among them. As for the constituents of medium, it was determined that the addition of fructose as a carbon source, malt extract as a nitrogen source, and metal salts, such as Mg, Fe, and Cu salts, raised antibiotic activity in vitro by 15 times. By post-testing, inhibiting effects on rice plant shirobakterebyo were identified.

Degradation of Trichloroethylene by Immobilized Cells

906C3826A Tokyo NIHON HAKKO KOGAKKAI
TAIKAI in Japanese 11-13 Oct 89 p 126

[Report by Hiroo Uchiyama, Kazuhito Oguri, Osami Yagi, and Etsuo Kouda, National Institute for Environmental Studies, Institute of Applied Biochemistry, University of Tsukuba]

[Text] 1. Purpose

Trichloroethylene is a nondegradable organic chloride used as a de-greasing detergent for metals and mechanical parts. It is carcinogenic and poses a problem as an underwater pollutant. The authors obtained a mixed culture system degrading TCE aerobically. They isolated a methanotrophe strain from this system. In this research, they studied various immobilizing agents and the concentrations required for degrading in order to apply this strain for environmental clarification as an immobilized reactor.

2. Method

As immobilizing agents, they applied high-molecular electrolytes (a complex of 30 mm of poly-vinyl potassium sulfate and 30 mm of diaryl-dimethyl ammonium chloride), 2 percent alginic acid, 2 percent carrageenan, and 3 percent agarose. Sixty ml of bacteria (1 mg/protein/ml) were collected, suspended in 2.5 ml of phosphate buffer solution, and immobilized by forming a sphere or a cube 3 mm in diameter with various immobilizing agents. The degrading experiment was performed as follows. Thirty ml of an inorganic medium and immobilized bacteria were sealed in a Bayer tube of 150 ml volume. TCE in various concentrations was

added, and the tube containing bacteria was stirred as a resting strain. The amount of TCE was quantitatively measured by gas chromatography according to the Head Space Method [phonetic].

3. Results

Seventeen hours after the reaction, the TCE of the initial concentration of 1 ppm was degraded 40 percent by un-immobilized bacteria, 13 percent by bacteria immobilized by high-molecular electrolytes, 91 percent by alginic acid, 66 percent by carrageenan, and 70 percent by agarose. These immobilized bacteria were reused after being reactivated by methane or methanol. As a result, we determined that bacteria reactivated by methanol degraded TCE a little better than others. Bacteria immobilized by alginic acid can degrade TCE even at 10 ppm, and they maintain the degrading activity when it was repeated three times.

Biotechnology of Limestone, Abalone Shell—in Vitro Test

906C3831A Tokyo NIHON BAIOMATERIARU GAKKAI TAIKAI in Japanese 27-28 Oct 89 p 12

[Article by Chikara Ohtsuki, Yukio Aoki, and Tadashi Kokubo, Institute for Chemical Research, Kyoto University; and Yoshitsugu Fujita, Seiya Kotani, and Takao Yamamuro, Department of Orthopedic Surgery, Faculty of Medicine, Kyoto University]

[Text] 1. Introduction

Limestone, the major component of which is CaCO_3 , consists mainly of calcite. Walker, et al., has reported that it combines with bone. In this research, in order to survey its combination mechanism, limestone was steeped into artificial humor having an inorganic ionic concentration nearly equal to human plasma, and the change in its surface structure was examined and compared with that of crystallized glass A-W. While abalone shell, the major component of which is also CaCO_3 , but which has a crystal structure different from limestone, was also steeped into the same artificial humor. The change in its surface structure was examined and the results were compared with those obtained from limestone.

2. Experiment

A 15x10x1 mm sample plate of limestone produced from Mina-city in Yamaguchi Prefecture, and a 15x10x0.5 mm sample plate of abalone shell were cut out, subjected to mirror grinding, and used as experimental materials. By X-ray diffraction analysis only calcite from limestone and aragonite from abalone shell were detected as crystal phases. As a result of thermal weight analysis it was learned that abalone shell contains about 4 weight percent organic substances. Each sample plate was steeped into artificial humor containing 142.0 mm of Na^+ , 5.0 mm K^+ , 1.5 mm Mg^{2+} , 2.5 mm Ca^{2+} , 147.8 mm Cl^- , 4.2 mm HCO_3^- , 1.0 mm HPO_4^{2-} , and 0.5 mm SO_4^{2-} at

36.5°C (pH was maintained at 7.25 by Tris buffer solution containing 50 mm of $(\text{CH}_2\text{OH})_3\text{CNH}_2$, and 45 mm of HC1). After 7-120 days, the change in surface structure was surveyed by X-ray diffraction and FT-IR reflection spectroscopic analysis.

3. Results

Limestone showed no change in surface structure when it was steeped in the artificial humor for 120 days. As for abalone shell, a thin layer of apatite was formed on the surface within 7 days, and the layer grew thicker as the period of steeping became longer. In case of crystallized glass A-W, apatite is formed on its surface within 7 days, and the crystallized glass combines with bone through the apatite. Therefore, the bonding of limestone to bone may be different from that of A-W, or the apatite formed on the surface may be extremely thin. Although, abalone shell forms a thin layer of apatite on its surface in the artificial humor, it does not combine with bone. This may be because organic substances contained in abalone shell inhibit the bonding.

Bonding Behavior of Limestone, Abalone Shell to Bone Examined

906C3831B Tokyo NIHON BAIOMATERIARU GAKKAI TAIKAI in Japanese 27-28 Oct 89 p 13

[Article by Yoshitsugu Fujita, Takao Yamamuro, Takashi Nakamura, and Seiya Kotani, Department of Orthopedic Surgery, Faculty of Medicine, Kyoto University; Tadashi Kokubo and Chikara Ohtsuki, Laboratory of Ceramic Chemistry, Institute for Chemical Research, Kyoto University]

[Text] Purpose

Generally, limestone consists of calcite, an ore, the major component of which is CaCO_3 . In 1983, Walker, et al., reported the bonding between bone and calcite and proposed a hypothesis on the bonding mechanism. This time, we have studied physically and chemically the bonding between bone and limestone containing nearly pure calcite, as well as its histological observations. The same study was conducted on aragonite which contains CaCO_3 as its major component, but the crystal structure of which is different from calcite, and abalone shell containing a small quantity of organic substances. The results have been compared with those obtained on the calcite.

Method

A plate of 15x10x2 mm was cut out from limestone containing nearly pure fine granular calcite, and was inserted under the skin of an adult rabbit. Eight weeks after this procedure, the rabbit was killed and non-deashed hard tissue preparations were produced. This was observed by Giemsa surface staining and CMR. The borders between the surface of the limestone and bone

were observed by SEM-EPMA. Eight weeks after the operation, a detaching test was conducted, and the failure load measured.

A plate of 15x10x0.5 mm was produced from abalone shell which was passed through an adult rabbit's shinbone in the same way. Non-deashed shinbone preparations were produced and observed with CMR. Then analysis by SEM-EPMA was performed. On the abalone shell, deashed tissue preparations were formed and stained with H.E.

Results

Eight weeks after the operation, the bonding between limestone and shinbone was very good; nearly the entire surface of the limestone was covered with new bone. By SEM-EPMA, limestone and bone were directly combined, but the reaction phase that was observed in other bioactive glass ceramics was not identified on the border. As a result of the detaching test, the average value of the failure load was 4.4 kg, which was less than the 7.4 kg average in the case of apatite-wollastonite containing glass ceramics. But it was nearly the same as the 4.3 kg average value of (ceravital) type crystalline glass KGS. A partial breakdown of limestone was identified on the dissociation face. Therefore, a problem remains with the strength of the material. But it can be expected to be used in orthopedic surgery as a bone-filling material. It is also an interesting material to study the bonding mechanism with bone.

On abalone shell, a clear zone was observed with CMR all around the material. It did not combine with bone, but, with SEM-EPMA, 5 μ m of Ca-P rich layer was observed. It is identified with H.E. staining that a connective tissue distinctive of penetration of inflammatory cells surrounds the abalone shell. It was surmised that foreign substance reactions by organic compounds contained in abalone shell prevented its bonding with bone despite the formation of Ca-P rich layers.

Bioactivity Change of CaO/SiO₂ Glass by Adding Various Ions Reported

906C3831C Tokyo NIHON BAIOMATERIARU GAKKAI TAIKAI in Japanese 27-28 Oct 89 p 14

[Article by Koichiro Oura, Takashi Nakamura, and Taka Yamamoto, Department of Orthopedic Surgery, Kyoto University; Takashi Kokubo, Institute for Chemical Research, Kyoto University; Yukihiko Ebisawa, Sumitomo Metal Industry; Yoshihiko Kotoura and Masanori Oka, Research Center for Medical Polymers and Biomaterials]

[Text] Purpose

We have previously reported that glass containing only CaO and SiO₂ produced apatite layer on its surface as Ca and Si eluted from glass react with ions in humor, and it combines directly with bone. Crystallized glass A/W-GC containing apatite and wollastonite crystals forms apatite as Ca and Si

eluded from wollastonite layer and glass layer react with ions in humor. Their bioactivity is determined by the reactivity of the glass layer which changes with the kinds and the amounts of ions added. This time, we surveyed the change in bioactivity of glass caused by the ions mixed in the glass layer by adding a trace of various ions to the glass containing CaO and SiO₂.

Method

Three weight percent of P₂O₅, Na₂O, B₂O₃, CaF₂, Al₂O₃ or Fe₂O₃ was added to glass consisting of 50 weight percent of CaO and SiO₂, with which a plate of 15x10x1 mm was formed. It was inserted into the shinbones of adult rabbits. The rabbits were killed 8 to 25 weeks after the operation. The changes which occurred on the glass surface were observed with CMR, Giemsa surface staining, and SEM-EPMA. The bonding strength with the bone was measured by a detaching test devised by Nakamura.

Results

Eight weeks after the operation, all plates, except for one consisting of glass which contained Fe₂O₃, formed a silica layer and an apatite layer on their surface and directly combined with bone. The surface of the glass containing Na₂O was weak. The glass containing Al₂O₃ formed incomplete apatite. The bonding of these two kinds of glass was weak. The glass containing P₂O₅, B₂O₃, or CaF₂ combined firmly with bone. Twenty-five weeks after the operation, the glass containing P₂O₅ was dissolved and bonding was weakened. The bonding of the glass containing CaF₂ was also weakened as the amount of bone to which glass adheres decreased. Only the glass containing B₂O₃ maintained strong bonding. The solubility of glass containing Fe₂O₃ was too low to form a reaction layer.

Discussion

This time the experiments were conducted with glass containing 3 weight percent of additives. Based on these results, the reactivity can be changed by changing the amount of additives, and by combining several additives. In case of A/W-GC, apatite does not have any relation to the bonding of bone. If magnetite or zirconia is deposited, crystallized glass can be magnetized or made stronger. The ions mixed into the glass layers determine bioactivity. If the kinds and amounts of ions are controlled, it becomes possible to produce crystallized glass that can meet the purpose of usage.

Adhesion Strength of Cultured Cells to Bioceramics Examined

906C3831D Tokyo NIHON BAIOMATERIARU GAKKAI TAIKAI in Japanese 27-28 Oct 89 p 31

[Article by Takashi Ushida, Tetsuya Tateishi, and Nobuhiro Moriya, Biomechanics Division, Mechanical Engineering Laboratory, Agency of Industrial Science and Technology]

[Text] 1. Introduction

Problems on the interface between a biomaterial and cells have been attracting attention recently. In this

situation, in order to solve the mechanism of cell adhesion to materials, an experiment to evaluate the adhesion strength of cells to materials was conducted by loading cells with grinding stress. As a result, it has been suggested that the adhesion strength of cells differ with physical and chemical properties of materials. In this research, the quantitative measurement of adhesion strength of cells was studied.

2. Cells

A connective tissue derived cell line of mouse (L-929) and a human osteosarcoma derived cell line (MG-63) were used as cultured cells. They were cultured in Eagle's MEM culture solution with 10 percent FBS added under the ambience of 37°C and 5 percent CO₂. Four days after the culture the cells were sowed on biomaterials. Three days after sowing, they were used for the experiment.

3. Materials

Disks (30 mm in diameter, 1 mm in thickness) of sintered hydroxyapatite (Mitsubishi Kogyo Semento), alumina (Kyosera), and zirconia (Nihon Tokushu Yogyo). The surface of these materials were abraded in order to control the surface roughness (Ra=0.1), and they were used as experimental materials.

4. Equipment

Tools were installed to a medium and low speed centrifuge (Hitachi Koki). The cells were loaded with stress perpendicular to the material surface caused by centrifugal power originated from the masses of cells. The experiment was conducted in the condition that materials and adhesive cells were touching the culture solution at 22°C.

5. Results

Under stress loading for a certain period of time (5 minutes), the adhesive rate of cells after stress loading differs distinctively with the kinds of cells and materials. In all experiments in this research, the adhesion strength of L-929 originated from connective tissue was smaller than that of MG-63 derived from osteosarcoma. As for one cell line, the adhesion strength of cells to HAP was significantly different from that to alumina or zirconia (HAP<zirconia or alumina). The extension of cells on the materials, or the adhesion area, cannot be defined generally, as the size of cells are different between L-929 and MG-63 (MG-63L-929). But, the adhesion area of MG-63 was larger than L-929.

The adhesion area of MG-63 to HAP was very different from that to alumina (HAP<alumina). (MG-63 was not a cell derived from a normal tissue, but this relationship between the cells derived from bone and HAP, one of bone components, is interesting). It is considered that there is no direct relationship between the adhesion strength and the adhesion area of cells, because the adhesion area of L-929 to HAP, and to alumina was nearly the same, but its adhesion strength to them was very different. The contact between cells and materials is

point-contact, but not face-contact. It is considered that adhesion occurs by interaction between fibronectin adsorbed in the materials and fibronectin receptors locally present at adhesion areas. In order to adhere cells directly to materials, MG-63 was treated with trypsin, cleansed with a sufficient amount of EMEM, and sowed on the materials. Then, they were cultured in MEM without fetal bovine serum, or MEM not containing plasma fibronectin, and their adhesion strength was measured. As a result, it was found that the adhesion strength was smaller than that obtained when cells were cultured in an FBS added medium, and there was no difference among materials. The mass was calculated from the volume of one cell, then its centrifugal power was calculated from the mass and acceleration, and 50 percent adhesion rate was determined as the adhesion strength of a cell. Thus, the adhesion strength was quantitatively defined. The adhesion strength of L-929 to HAP was calculated up to 10⁻¹⁰N.

Influence of Trypsin on Adhesive Behavior of Primary, Transformed Osteoblasts Studied 906C3831E Tokyo NIHON BAIOMATERIARU GAKKAI TAIKAI in Japanese 27-28 Oct 89 p 33

[Article by Richard Michael Shelton, Tetsuaki Matsuda, and Mikinori Ogura, Dental School, Kagoshima University]

[Text] Introduction

In order to survey cell reactions to biomaterials it is a general approach to use cells separated by treating them with enzymes *in vitro*. In this research, we studied the influence of trypsin on the adhesive behavior of both primary and transformed osteoblasts.

Materials and Method

Three kinds of culture vessels were used: tissue culture dishes (TC), bacteriological dishes (BAC), and BAC dishes where one-half of their content was treated with concentrated sulfuric acid^{1,2} and the other half was not.

The parietal bone of an infant rat with its periosteum removed was finely ground and cultured in the three kinds of dishes to produce the primary osteoblasts without trypsin treatment. In order to produce the primary osteoblasts with trypsin treatment, a parietal bone was treated with 0.05 percent trypsin solution at 25°C for 10 minutes, and the cells were dispersed. Transformed osteoblasts were produced by using MC3T3,³ and by treating it with trypsin in the same way. Then the treated cells were dispersed. The same number of these two cells treated with trypsin were inoculated in the three kinds of dishes. α-MEM was applied as the medium. The cells were then cultured at 37°C with 5 percent CO₂.

Results

In the TC dishes, the three kinds of cells adhered very well. The number of floating cells were highest in the primary osteoblasts with trypsin treatment.

In the BAC dishes, the three kinds of cells adhered very well in the area treated with acid. The primary osteoblasts formed colonies in the area treated with acid, and showed well-expanded forms. At the border with the nontreated area, cells arranged parallel to the border (haptotaxis⁴). In the nontreated area, cells were spherical, but retained colony-forming ability. The primary osteoblasts with trypsin treatment did not adhere in the area which was not treated with acid, and had no colony-forming ability. MC3T3 cells formed colonies in the untreated area, but cells turned spherical and the colonies coagrated gradually. At the border, the thigmotaxis of cells in the treated area was very weak at the primary stage, but it grew stronger as time passed.

Conclusion

1. The primary osteoblasts were more sensitive to trypsin injuries than the transformed cells.
2. The adherability of the primary osteoblasts at the initial stage was different from that of the primary osteoblasts with trypsin treatment and transformed MC3T3 cells.

3. In the experiment on the colony formation against biomaterials, it was suggested that sufficient care should be taken to translate the results obtained from the primary and the transformed osteoblasts with trypsin treatment.

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**Demonstration Irradiation of MOX Fuel in
Mihama Unit-1**
*90FE0115A Tokyo ATOMIC ENERGY SOCIETY OF
JAPAN in Japanese 2 Apr 90 p 176*

[Presentation by Yoshinori Matsuoka, Tatsuo Nagoh, Shinichi Nakagawa, Hiroshi Tochihara, Kazuya Seki and Toshikazu Ida of Kansai Electric Power Company, Ltd.; at the 1990 conference of the Atomic Energy Society of Japan, 2-4 April 1990, at Tokyo University]

[Text] Demonstration Irradiation of MOX Fuel in Mihama Unit-1

1. Foreword

Use of plutonium in light water reactors (LWR) is being considered in order to conserve uranium resources and to establish plutonium use technology needed for fast breeders in the future. In LWRs, plutonium is used in the form of uranium-plutonium mixed oxide (MOX) fuel, but before full-scale employment of this MOX fuel, four rods of the MOX fuel initially are being loaded and irradiated in Mihama Unit-1 (a pressurized water reactor (PWR) of Kansai Electric Power Company, Ltd.) in a demonstration irradiation using a small number of fuel rods. (Irradiation began in April 1988; the first cycle ended in March 1989; and the second cycle began in August 1989.)

The purpose of this research was to follow the combustion of the MOX fuel and to confirm the appropriateness of nuclear design for MOX fuel.

2. Design of Aggregate MOX Fuel

The specifications for aggregate MOX fuel excluding fuel material, is virtually identical to 14 x 14 type UO₂ used in Mihama Unit-1. Also, the mechanical design of the aggregate MOX fuel, nuclear, thermal and hydropower design criteria, and the characteristics of the reactor core used are designed to be equal to UO₂ fuel. Additionally, as shown in figure 1, this MOX fuel is designed to suppress output peaks within the aggregate by having three types of PuO₂ mix ratio distribution. Now, the aggregate average PuO₂ mix ratio is about 3.8 wt%, and in terms of reactivity, it is equivalent to 4 wt% UO₂ fuel.

**3. Comparison of Measured Value and Design Value in
Mihama Unit-1 Seventh Cycle**

Table 1 is a comparison of design value and measured value resulting from physical testing during start-up of the

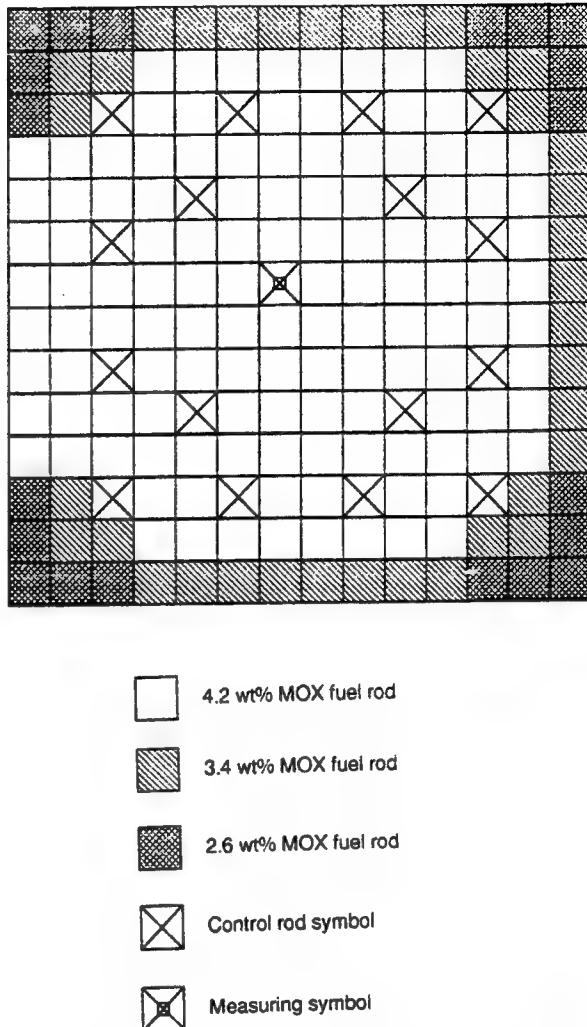


Figure 1. Aggregate MOX Fuel Mix Ratio Distribution

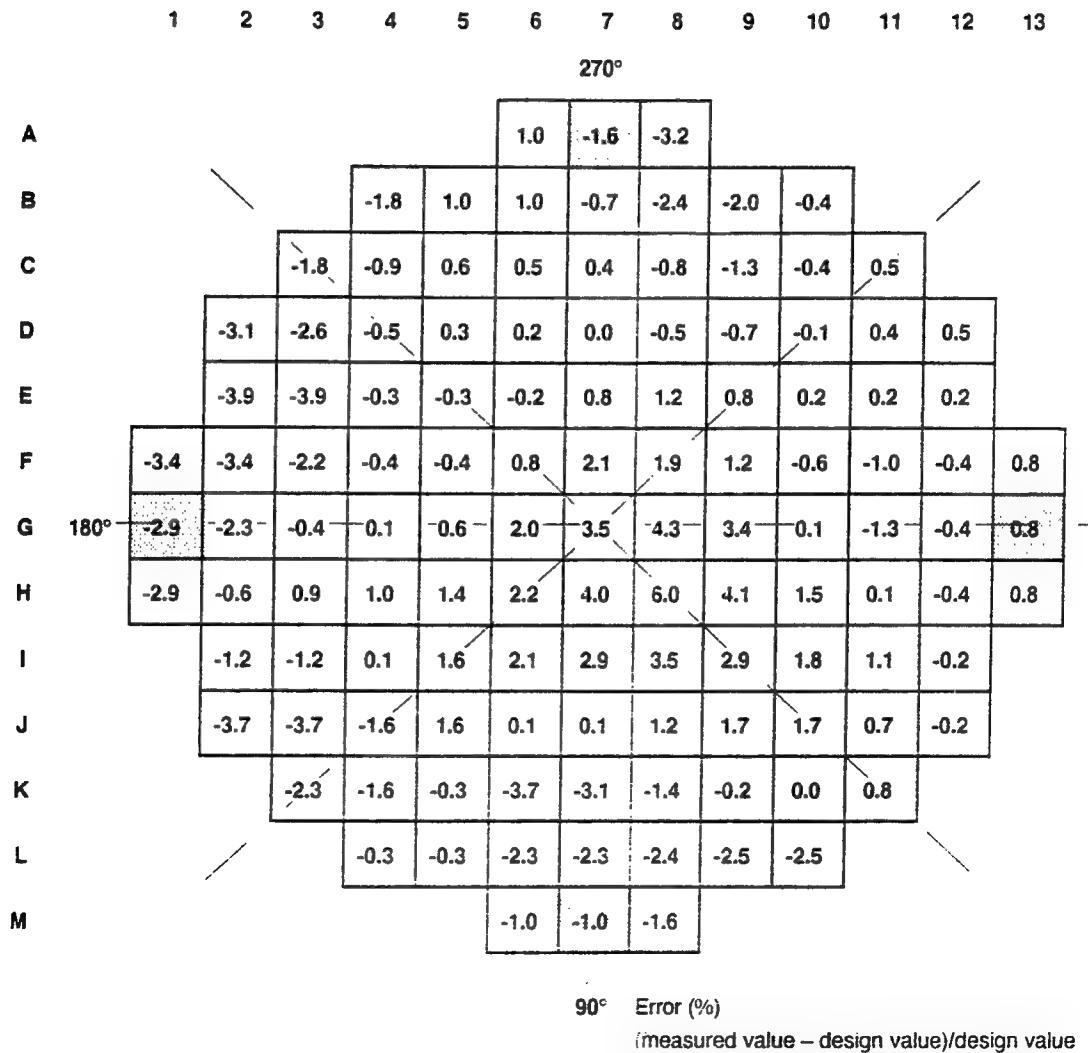
reactor in the seventh cycle (first irradiation cycle) of Mihama Unit-1. Figure 2 shows the results of output distribution processing during times of high-temperature and zero output. In both, measured value and design value show a favorable consistency. In figure 3, the aggregate relative output from aggregate MOX fuel is plotted throughout the cycle. In this, no uniqueness was observed in the aggregate MOX fuel, and it was found that the method of nuclear design was appropriate for MOX fuel.

Conclusion

Four aggregate MOX fuel rods were loaded and irradiated in Mihama Unit-1. It was confirmed, as a result of physical testing of the reactor during start-up and by following data of the operation of the reactor, that there is good consistency between measured value and design

value, which confirmed the appropriateness of the nuclear design method for MOX fuel.

The four aggregate MOX fuel rods are in a second irradiation cycle, and verification of the appropriateness of the nuclear design method when combustion has advanced will be conducted by continuing to follow the rods in the state of combustion.



**Figure 2. Percentage Error of Design Value to Measured Value of Output Distribution
(at start of reactor core life; high temperature zero output)**

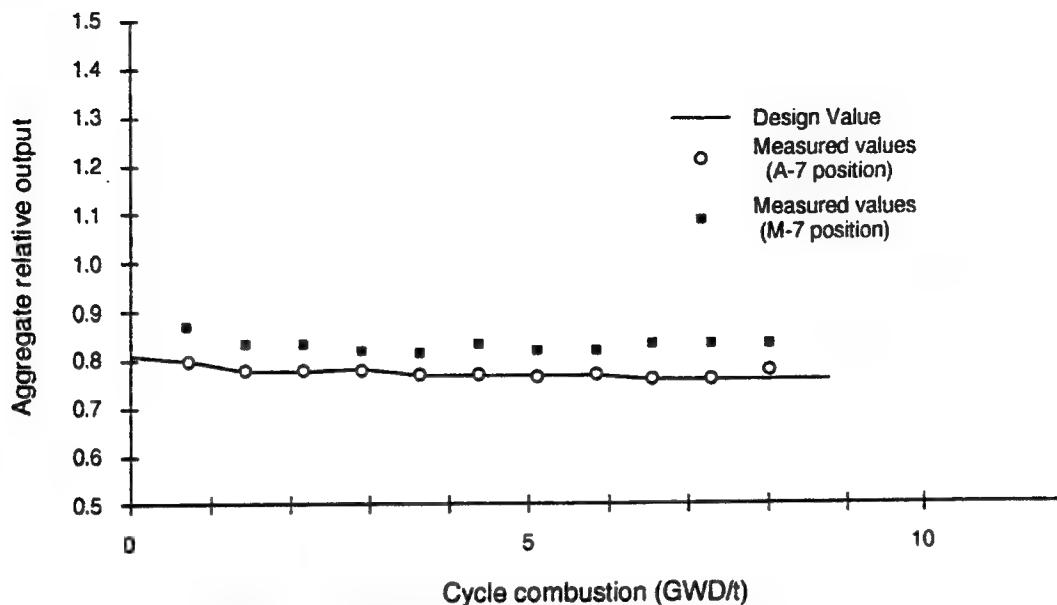


Figure 3. Change Accompanying Aggregate Output Combustion

Item	Design Value (P)	Measured Value (M)	Error (M-P)	Criteria for decision to reevaluate
Critical Boron Temperature	All control rods removed	1,237 ppm	(M-P) -4 ppm	(M-P) \pm 50 ppm
	Bank B for control fully inserted	978 ppm	(M-P) -5 ppm	Same as above
	Bank A for control fully inserted	792 ppm	(M-P) -2 ppm	Same as above
Moderator Temperature Coefficient	All control rods removed	-1.0 $\text{pcm}^{\circ}\text{C}^{-1}$	(M-P) -2.3 $\text{pcm}^{\circ}\text{C}$	(M-P) \pm 3.6 $\text{pcm}^{\circ}\text{C}$
Control Rod Value	Bank B for control	2,448 pcm	(M-P)/P -6.8%	(M-P)/P \pm 10%
	Bank A for control	1,785 pcm	(M-P)/P -7.5%	Same as above

$\text{pcm} = 10^{-5} \Delta k/k$

Table 1. Results of Physical Test of Reactor During High-temperature Zero Output

Status of R&D of Plant Aging, Plant Life Extension

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[Presentation by Shiori Ishino of the Engineering Department, Tokyo University, at the 1990 conference of the Atomic Energy Society of Japan, 2-4 April 1990, at Tokyo University]

[Text] Status of R&D of Plant Aging, Plant Life Extension

1. Foreword

Recently, a great deal of interest has become focused on plant aging and plant life extension of nuclear power plants. This presentation will be limited to light water reactor (LWR) nuclear plants, which already have a

history of 30 years of commercial use. In the United States, by the year 2000, there will be LWRs with an original design service period of 40 years. For this reason, it is thought that the ratio of nuclear power generation will go down substantially in the next century, from about 2010. In Japan, a series of LWRs have been built since 1970, and at present, 39 nuclear power plants are in operation, including those in trial run mode and one gas reactor. Japan will face the same situation as the United States, only 10 years later. In the United States, research began in 1978 by the Electric Power Research Institute (EPRI). In Japan, the power facilities technology inspection association (Hatsuden Giken) began an 8-year R&D program in 1985 under the auspices of the national government. R&D also is being conducted by a number of research institutes and electric power companies. This presentation first of all will review that status of domestic and foreign R&D efforts.

2. Content of R&D on Plant Aging and Plant Life Extension

Plant aging and plant life extension form an indivisible pair. The content of research can be divided into four areas:

- (1) Selection of factors that determine plant life: Selection of equipment to be covered by R&D
- (2) Plant life evaluation and estimation technology
- (3) Plant aging diagnosis and monitoring technology
- (4) Replacement and repair technology

In the final analysis, most of the R&D involves changes over a long period of time of the behavior and characteristics of the component materials that make up a power plant. This naturally involves predicting an

unknown future from existing data bases. I would like to point out that since a study tracing the features of degradation of equipment is needed in order to increase reliability of prediction, research covering a rather broad range, from fundamentals to application, is necessary.

3. Selection of Equipment and Status of R&D

As stated earlier, selection of components that contribute to the determination of plant life is needed in research on plant aging and plant life extension. In the above-mentioned R&D by Hatsuden Giken, critical equipment for pressurized water reactors (PWR) and boiling water reactors (BWR) in Phase I (FY85-86), as shown in the chart, were selected, and a preliminary evaluation of plant life was carried out. The main

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	FY85	FY86	FY87	FY88	FY89	FY90	FY91	FY92
(1) Nuclear Power Plant/ Facilities and Equipment								
1. Feasibility Survey		Phase I Feasibility Survey						
2. Verification Test		Step 1: • Survey of current technology	Step 2: • Selection of equipment to be researched • Preliminary evaluation of plant life extension • Basic plan for verification test					
				Phase II Verification Test				
				• Testing of low alloy steel and stainless steel in corrosive environment • Thermalization time efficiency test of two-phase stainless steel • Stainless steel irradiation fracture toughness test • Stainless Steel irradiation SCC test • Regeneration confirmation test: reactor pressure vessel surveillance test • Inspection of reactor pressure vessel/in-pile structure; development of repair equipment				
3. Plant Life Extension Technology Evaluation				Phase II Plant Life Extension Technology Evaluation				
				Step 1: • Plant life extension concept in brief • Draft Data Format • Collect existing data	Step 2: • Plant life extension evaluation plan • Study of life estimate methods • Study of aging diagnosis, monitoring evaluation methods • Collect existing data	Step 3: Same as Step 2	Step 4: • Life estimate evaluation • Aging diagnosis; monitoring evaluation; system program • Large-scale equipment replacement • Repair evaluation; concept design	Step 5: Same as Step 4
(2) Nuclear Power Plant/ Concrete Structure								
1. Feasibility Survey		Phase I Feasibility Survey						
2. Plant Life Extension Technology Evaluation		Step 1: • Survey of current technology	Step 2: • Durability evaluation					
				Phase II Plant Life Extension Technology Evaluation				
				Step 1: • Basic study plan • Life estimate in brief • Plant life extension evaluation	Step 2: Same as Step 1			
(3) Overall Evaluation								
					Phase III Overall Evaluation			
					Step 1: • Economic evaluation in brief • Regulations and standards study	Step 2: • Economic evaluation • Regulations and standards study	Step 3: • Overall evaluation of plant life extension • Life estimation methods • Plant aging data • Nuclear power plant life extension plan • Topics for the future	

Light Water Reactor Plant Life Extension Technology Development Project Underway in Power Facilities
Technology Inspection Association

equipment selected included reactor pressure vessel, in-pile structure, steam generator (SG), pressurizer, primary coolant system piping and pump, and containment vessel.

When the scheduled in-service periods of these components are extended, some factors that will cause degradation of neutrons and other materials can be considered, such as radiation exposure, stress corrosion, corrosion fatigue, thermalization time efficiency degradation and general corrosion. Of these, the following four items were extracted as being the most critical, and verification tests have begun:

- (1) Characteristics of corrosion of low alloy steel and stainless steel: Collection of data on fatigue in corrosive environments, using degradation of thermalization time efficiency on simulation materials A533 and A508 steel as well as SUS304 and 316 steel; and development of preliminary model
- (2) Post-irradiation fracture toughness of stainless steel: Collection of data on the decline of toughness of actual in-pile material austenitic stainless steel (SUS304, 316) due to irradiation; and development of preliminary model
- (3) Stainless steel stress corrosion cracking (SCC) under irradiation: Clarification of neutron irradiation affect on SCC (IASCC); and development of preliminary model
- (4) Thermalization time efficiency of two-phase stainless steel: Development of preliminary model, through the use of simulation materials, of the decline of toughness due to degradation of thermalization time efficiency of two-phase austenitic/ferrite stainless steel (A351, CF8, CF8M, etc.) that is used for primary coolant system piping, pump casing, etc.

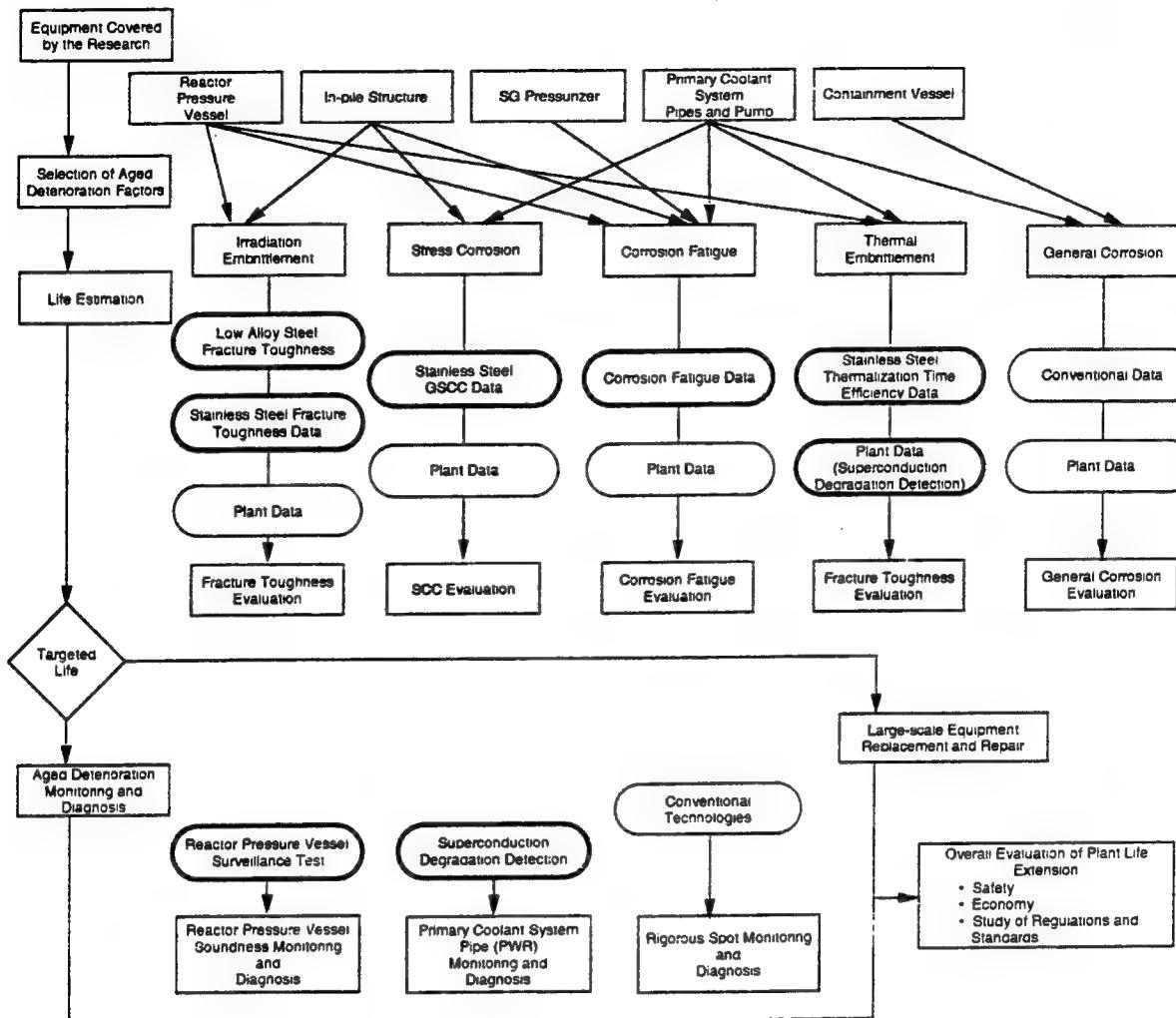
These are important issues not only for LWRs but other nuclear power plants as well, and the results of research in progress is being awaited.

With respect to fracture toughness of low alloy steel, Hatsuden Giken also is conducting research concerning the problem of pressure vessel thermal shock under pressure, and some results have been made public. In this case as well, the establishment of a preliminary model based on the features of decline of toughness is important, and an international working group has been formed for this task. This presentation will touch upon the features of decline of toughness of pressure vessels and the hypothesized features of IASCC.

4. Study and Conclusion

This review has addressed some of the R&D on plant aging and plant life extension, but it has foregone discussion of monitoring as well as replacement and repair technology. For pressure vessels, research has been conducted for many years on recovery of toughness through annealing, but the stage of practical application has not yet been reached.

Now, the mainstream of present research on materials irradiation, which forms the base of this R&D project, has been conducted under simple conditions, and the results of this project necessarily reflect that fact. However, as can be seen in a most extreme example—the fact that creep during irradiation and creep after irradiation are completely separate phenomena, the correctness of evaluation of a phenomenon during irradiation by means of tests after irradiation must be substantiated by structural theory. There are instances where combining the ways that a material behaves in response to single conditions will not be the same as the way it behaves in response to multiple conditions. It is in this area that it is hoped that researchers conducting basic research in the universities will contribute.



Flow Chart for Selection of Items for Verification Test

5. In Conclusion

This presentation owes much to the report of the LWR plant life extension documentation research working group of the "neutron irradiation damage evaluation" research committee, Atomic Energy Society of Japan

(AESJ) (AESJ Journal 30, nr. 9, 1988, p 13-25). I also would like to express thanks to department director Tatsuo Kawakami and Mr. Takeshi Matsuzaka of Hatsuden Giken for the materials that they gave me on recent trends.

Committee for the Study of IMS International Joint Research Program

906C3845A Tokyo SEIFU SHIRYO-TO FUKYU CHOSA-KAI in Japanese 30 Mar 90 pp 19-25

[Article by the Industrial Machinery Division, Machinery and Information Industries Bureau, Ministry of International Trade and Industry]

[Text] 1. Objective of This Committee

With increasing changes in the social environment, the manufacturing industry in the advanced industrial countries are facing various problems that could endanger the very foundation on which it stands. For example, the absolute numbers of craftsmen and skilled laborers are in short supply, and moreover as their average ages and their educational levels increase, the laborers are showing a tendency to desert the manufacturing industry in favor of the tertiary industry because of the work hours and the work environment. The problems besetting the manufacturing industry are shared by all the advanced industrial countries, and they need to be solved by the cooperative efforts of all advanced industrial countries. If the manufacturing industry is to see a healthy growth in the world economy, development of technologies that will enable these problems to be solved and will facilitate the growth of an attractive manufacturing industry is an essential condition.

In light of such a situation, there is abundant room for Japan to contribute to the healthy growth of the world's manufacturing industry. The economic growth of Japan after World War II could not have been achieved without technology transfers from the United States and Europe. We believe that Japan, which boasts excellent achievements to its credit in production technology today, should embark on the development of new technology ahead of others and take the initiative in disseminating the achievements for the common benefit of the world. To that end, this committee will prepare a platform as the foundation for proposing the institution of an international joint research program (IMS international joint research program) for the 21st Century that will find wide application in the world, and at the same time will work to win the consensus of Japan, North America, and Europe so that the program will be realized.

2. Contents of the Report

(1) Concept of IMS

At present, the manufacturing industry of Japan is suffering from shortages in the absolute numbers of craftsmen and skilled workers. Furthermore, with increases in the average ages of workers and in their educational levels, they are increasingly seeking employment in the tertiary industry, thereby leading to the so-called "deserting the manufacturing industry" phenomenon among engineers and engineering and science students. Thus, the situation that could endanger the

very foundation on which the manufacturing industry stands has manifested itself in part of the industry.

The situation is the same in the advanced countries—the United States and European countries—and it is identified as a common grave problem.

The so-called IMS (intelligent manufacturing system) is an advanced production system for the 21st Century that will overcome such problems and contribute to the growth of an attractive manufacturing industry, the very backbone of a country's economic activity, by maintaining and enhancing its vitality.

(2) Definition of IMS

IMS is an advanced production system geared to the 21st Century that will find wide use in the world and is defined as follows: "While taking advantage of various intelligent activities in the manufacturing industry and attempting to bring about harmony between intelligent machines and humans, all corporate activities ranging from the acceptance of an order to its design, production and marketing will be flexibly fused and managed in order to raise productivity.

(3) Outline of IMS International Joint Research Program

A. Contents of R&D

In order to establish IMS technology, the following research and development will be undertaken.

- (a) To improve and systematize the existing and current-use technologies so that they will be used by both the advanced countries and NIES.
- (b) To standardize the existing and next-generation technologies.
- (c) To research and develop new, advanced production systems geared to the 21st Century.

B. Period

10 years.

C. Total Project Scale

About 150 billion yen (to be shouldered by the government and private sectors in Japan, North America and Europe)

D. R&D Structure

In order to promote this project, the "International IMS Promotion Organization (provisional name)" will be established in either the United States or Europe.

Within the organization will be established an international joint research institute where researchers on loan from various countries (universities, research institutes, private enterprises) will undertake joint research, and it

will also award research groups made up of existing universities, and enterprises with contract research projects.

E. Research Fields

The fields targeted for research are the following, and R&D will be conducted within several projects. The scale of a project is 1 - 1.5 billion yen, to be carried out in a three to five year period, and 100 projects will be undertaken over a 10-year period.

(a) Production System Construction Technology

To establish the concepts and methods that will be needed for building the IMS systematically and on a common ground.

(b) Data and Communications Technology Pertaining to Production

To develop the data and communications technology as the infrastructure for IMS.

(c) Production and Control Apparatus and Machining Technology

To optimize the manufacturing and control apparatus comprising the IMS and to provide them with intelligent capabilities.

(d) Advanced Materials Application Technology

R&D of new materials that will help to drastically upgrade the manufacturing equipment.

(e) Human Factors Pertaining to Humans

R&D of social, economic, environmental and human factors related to IMS.

(4) How To Promote IMS (draft)

A. Committees

In order to draft 10-year plans for the IMS, at least the following two committees will have to be inaugurated in the initial year.

The first is the IMS international committee. Made up of high-level experts and researchers from a broad field of society, including industry, academia, and research institutes, it is the highest decision-making organ for IMS. The committee will discuss the basic policies for IMS, such as the concepts of IMS, how to promote R&D of IMS, and distribution of research achievements, etc., and will work for an international consensus to be achieved. The committee is to be made up of about an equal number of members from Japan, North America, and Europe.

The second is the technical assessment committee, which will evaluate all technical matters pertaining to IMS.

To be concrete, the committee will evaluate planning papers submitted by enterprises, universities, etc., will

organize international groups responsible for drafting conceptual designs for IMS, and will consolidate all of them into an entire body in a period of about one year in order to come up with a draft plan for the IMS 10-year program.

The committee will also be comprised of about an equal number of members from Japan, North America, and Europe.

B. Establishment of IMS Center

As an intermediate measure before the international IMS Promotion Organization (provisional name) is established, an IMS center is expected to be established, which will operate the aforementioned committees and function as the core organization for promoting the IMS program. Beside convening meetings of the IMS International Committee, the IMS Center will promote a grassroots campaign for the promotion of the IMS concept in Japan by holding, with the cooperation of the Regional Industrial Bureaus, IMS symposiums in various areas of the country.

C. Schedules

4 and 5 June: The first IMS International Committee meeting (in Tokyo).

30 June: The deadline for submission of the planning papers.

Early July: The planning papers will be forwarded to pertinent Technical Assessment Committees.

Mid-July-Late August: Based on the evaluations of the Technical Assessment Committees, six groups will be organized.

Early September: The work for conceptual designs will be contracted out.

Early October: The second IMS International Committee meeting (in the United States).

February 1991: The third IMS International Committee meeting (in Europe).

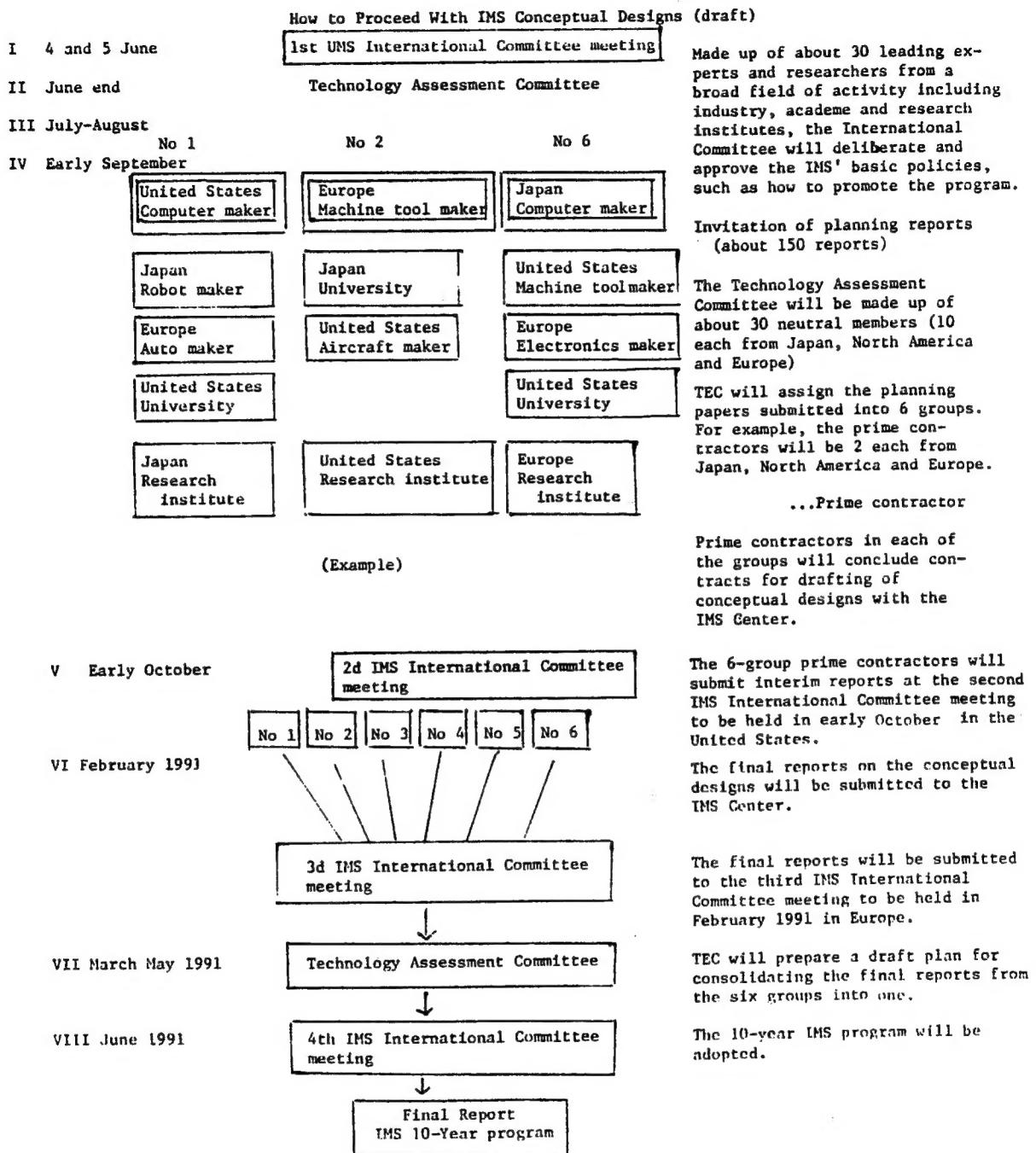
March-May 1991: The Technical Assessment Committees will consolidate the various IMS concepts into a single program.

June 1991: The fourth IMS International Committee meeting (the IMS 10-year program will be adopted).

Roster of Members on IMS International Joint Research Program Deliberation Committee

Chairman: Hiroyuki Yoshikawa, dean of the Faculty of Engineering, University of Tokyo

Vice chairman: Yuji Furukawa, professor, Mechanical Engineering, Faculty of Technology, Tokyo Metropolitan University



Members: Tamio Arai, professor, Precision Engineering, Faculty of Engineering, University of Tokyo; Yoshimi Ito, professor, Production Mechanical Engineering, Faculty of Science and Engineering, Tokyo Institute of Technology; Kazuaki Iwata, professor, Electronic Control and Mechanical Engineering, Faculty of Engineering, Osaka University; Sachio Hasegawa, professor,

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